

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

RECORD COPY  
CENTRAL FILE

(19) RÉPUBLIQUE FRANÇAISE  
—  
INSTITUT NATIONAL  
DE LA PROPRIÉTÉ INDUSTRIELLE  
—  
PARIS  
—

(11) N° de publication : 2 635 441  
(à valoir que pour les  
concessions de reproduction)

(21) N° d'enregistrement national : 88 10991

(51) Int. Cl. : A 23 G 300.

(12)

BREVET D'INVENTION

B1

(54) CONCENTRÉS DE GOMME DE BASE A HAUTE TENEUR EN POLYMERES, LEUR PROCÉDÉ DE PRÉPARATION ET LEUR UTILISATION DANS LA FABRICATION DE GOMME A MACHÉ

(22) Date de dépôt : 18.08.88.

(30) Priorité :

(60) Références à d'autres documents nationaux apparentés :

(71) Demandeur(s) : Société dite : GENERAL FOODS FRANCE. -FR.

(43) Date de la mise à disposition du public de la demande : 23.02.90 Bulletin 90/08.

(72) Inventeur(s) : FRANCOIS BOUDY

(45) Date de la mise à disposition du public du brevet d'invention : 27.12.91 Bulletin 91/52.

(56) Liste des documents cités dans le rapport de recherche :

(73) Titulaire(s) :

*Se reporter à la fin du présent fascicule*

(74) Mandataire(s) : CABINET BROT et JOLLY

FR 2 635 441 - B1



## PATENTS SUMMARY

04/23/99

Page 1

Country : France

Patent #: 2,635,441 B

Index#: 3279

Inventor: Francois Boudy

Issue Date: 12/27/91

Assignee: General Foods

Date of Application: 08/18/88

Title:

GUM BASE CONCENTRATES WITH HIGH POLYMER CONTENT, THEIR  
PREPARATION PROCESS  
AND THEIR USE IN CHEWING GUM PRODUCTION

Desc.:

The process for preparing gum base concentrate or premix, the product so produced and the use in gum are claimed. High molecular weight base components are ground into particles, mixed and plasticized. The mixture and fillers are added to an extruder in different ports. Top temperature is 120 degrees C and maximum time is 1.5 minutes.

Key Words:

10 CHEWING GUM  
350 GUM BASE  
352 Natural Rubbers  
353 Synthetic Rubbers  
361 Fillers  
371 Softeners/Plasticizers  
502 Base Manufacture/Preparation  
506 Extrusion  
564 Particle Size  
569 Improved or Easier Processing  
713 General Foods  
805 France

RECORD COPY  
CENTRAL FILE

GUM BASE CONCENTRATES WITH HIGH POLYMER CONTENT,  
THEIR PREPARATION PROCESS AND THEIR USE IN CHEWING  
GUM PRODUCTION [Concentrés de gomme de base à haute  
teneur en polymères, leur procédé de préparation et  
leur utilisation dans la fabrication de gomme à  
mâcher]

French 2,635,441

Translated from French

4444/WR

DH/rjs

10/23/90

3279

350

352

353

354

357

358

356

354

359

373

375

[19] France

National Institute of Industrial Property, Paris

[11] Publication No.: 2 635 441

[12] Patent Application A1

[21] National Registry No.: 88 10991

[22] Application Date: August 18, 1988

[30] Priority:

[41] Promulgation Date:

[43] Date Application Laid Open to Public  
Inspection: BOPI "Patents" No. 8 of  
February 23, 1990

[51] Int. Cl.: A 23 G 3/30

[60] Citation:

[71] Applicant: General Foods France

[72] Inventor: François Boudy

[73] Assignee:

[74] Representative: Cabinet Brot et Jolly

[54] Title: GUM BASE CONCENTRATES WITH HIGH  
POLYMER CONTENT, THEIR PREPARATION  
PROCESS AND THEIR USE IN CHEWING  
GUM PRODUCTION

[57] ABSTRACT

The invention relates to a process for preparing a gum base concentrate with a high polymer content of the type including high molecular weight elastomers, plasticizers and mineral fillers.

This process is characterized by the fact that the high molecular weight elastomers are ground into particles, these particles are mixed in the desired proportions and plasticized; the resulting mixture and the mineral fillers are introduced separately in the desired proportions in a twin-screw extruder 6 with sleeves 0, 1', 2', 3',

4', 5', in which the temperature does not exceed 120°C or the elastomer mixture does not remain more than about one minute and thirty seconds, and whose screw speed is less than about 120 rpm, and the concentrate exiting the extruder is recovered.

**GUM BASE CONCENTRATES WITH HIGH POLYMER CONTENT, THEIR PREPARATION  
PROCESS AND THEIR USE IN CHEWING GUM PRODUCTION**

The present invention relates to gum base concentrates with high polymer content which can be used in the preparation of gum base for chewing gums.

The invention also relates to the preparation of such concentrates and their use in the production of chewing gums.

It is known that gum bases normally include high molecular weight polymers, mineral fillers and plasticizers. High molecular weight polymers include, but are not limited to, isoprene-isobutylene copolymers, styrene, butadiene and polyisobutylene, while low molecular weight polymers include polyisobutylene and polyvinyl acetate. Plasticizers include resins of rosin and waxes.

Gum bases can be prepared directly by the chewing gum manufacturers. They can also be produced separately and be sold as semi-finished products.

They are usually produced in two stages.

In a first stage, a premix based on polymers, plasticizers and mineral fillers is prepared. This premix is then transferred into a mixer, where the rest of the usual components of the gum base are added. This second stage does not concern the present invention, and we will discuss in the following only the production and formulation of the premixes.

The premixes can be prepared by dissolving the main polymer at a high temperature in the other materials. This dissolving operation lasts about 3-6 hours and is performed in an open mixer with moveable blades called a "pug mill," in which the mixture is held between 120 and 150°C throughout the operation. This technique, however, has the drawback that the polymer undergoes degradations in the elastic chains due to the temperature at which

the dissolving is performed and due to the duration of a cycle. As a result, the product obtained has mediocre elasticity, whereas good elasticity is an important characteristic of a satisfactory premix.

The premix of the base polymer and the other materials can also be performed at constant volume and under pressure in a high-powered shearing mixer known as a "pug mill." The duration of a mixing cycle is 20-30 minutes, and the product undergoes considerable mechanical and thermal stresses.

This process, like the one described above, thus involves degradation of the elastic properties of the polymer. Furthermore, such a process cannot be applied to the preparation of tacky or viscous products, and it requires the resulting mixture to have a certain consistency.

Finally, the premix can be conducted by co-extruding the different components, but this process, which combines the effects of temperature, pressure and shearing, also involves degradation of the elastic properties of the polymer.

Regardless of the technique used to prepare these premixes, they never contained more than about 50% polymer by weight.

The present invention intends to remedy the drawbacks of the above-described prior art.

One purpose of the present invention is therefore to propose a process for preparing a concentrate with a high polymer content that can attain and exceed 85% by weight, which does not substantially alter the elastic properties of these polymers.

Another purpose of the invention is to propose a process that can be used without restrictions for preparing the concentrate with a high content of polymers, even viscous or tacky ones.



Another purpose of the invention is a concentrate whose polymer content can attain and exceed 85% by weight.

The invention also intends to propose a concentrate of this type that exhibits satisfactory elasticity for preparing chewing gums.

Finally, the invention aims to propose a concentrate such that industrialists can easily apply it to the preparation of their own products by adding to it the additives they normally use in the necessary proportions.

For this purpose, the object of the invention is a process for preparing a gum base concentrate with a high polymer content of the type including high molecular weight elastomers, plasticizers and mineral fillers, characterized by the fact that high molecular weight elastomers are ground to the form of particles; these particles and plasticizers are mixed in the desired proportions; the resulting mixture and the mineral fillers are introduced separately in the desired proportions in a twin-screw extruder with sleeves, in which the temperature does not exceed 120°C, in which the elastomer mixture does not spend more than about 1 min 30 sec, and whose screw speed is less than about 120 rpm, and the concentrate exiting the extruder is recovered.

In this definition of the process according to the invention, in the rest of the description and in the claims that follow, the term high molecular weight elastomers refers to elastomers having a numerical molecular weight of at least 220,000 and a molecular weight by weight of at least 450,000.

Advantageously, the mixture of high molecular weight elastomer and plasticizers will be introduced into the sleeve arranged furthest upstream in the twin-screw extruder, while the mineral fillers are introduced downstream, preferably in two different sleeves of the extruder to enhance their

distribution in the finished product. The particles of ground elastomer may have a size on the order of 5 mm.

In the event tacky materials such as low molecular weight elastomers must be present in the concentration thus prepared, they are introduced in the molten state into the twin-screw extruder downstream from the mixture of high molecular weight elastomers and plasticizers and upstream from the mineral fillers. By low molecular weight elastomers we mean elastomers having a molecular weight by number less than 4,000 and a molecular weight by weight less than 150,000.

The process in accordance with the invention therefore aims to avoid exposing the elastic chains of the elastomer to excessive temperature and shear stresses or subjecting them to such stresses for too long.

An embodiment of this process will be described in the following in greater detail as a non limiting example with reference to the attendant drawings. In these drawings:

Figure 1 is a schematic view of the apparatus used in this embodiment of the process;

Figures 2a, 2b and 2c are diagrams illustrating the conditions of operation of the five-sleeve extruder of this apparatus.

As we see in figure 1, the high molecular weight elastomer is first ground into particles of the same size of five millimeters in an impact disc mill 1. The ground product is then mixed in a ribbon mixer 2 with plasticizers (resins of rosin, waxes, etc) fed through line 3, and the resulting mixture is transferred into a vibrating weight metering hopper 4.

The mineral fillers to be added to the product (talc and calcium carbonate) are loaded into two vibrating weight metering hoppers 5.

These different metering hoppers then introduce the materials they contained in specific proportions into a twin-screw extruder 6, which in this case, is a "five-sleeve" extruder (recall that, in the usual classification of extruders, the input sleeve, or sleeve 0, is not taken into account; in the present installation, the extruder thus actually includes six sleeves 0, 1', 2', 3', 4' and 5'). More precisely, after preheating twin-screw extruder 6, while the screws are driven by the motor, the premix consisting of high molecular weight elastomer and plasticizers is fed into sleeve 0, or the input sleeve of the extruder, while the mineral fillers are introduced by the two metering hoppers 5 in two different sleeves, for example, sleeves 0 and 2', of the twin-screw extruder to improve the homogeneity of the finished product.

If the concentrate being prepared must contain low molecular weight elastomers, the latter are fed in the molten state into a tank 8, heated to a temperature of 100-120°C by oil circulating in a closed circuit 9 from a boiler 10. These elastomers are then injected by a gear pump 11 into sleeve 1'.

The entire assembly may, naturally, operate continuously.

The screws of extruder 6 may rotate in the same or different directions.

For a given flow rate, their rotational speed will be chosen such as to minimize degradation of the polymer while insuring satisfactory entry of the materials into the machine.

At the rate of 120 kg/hr, using a five-sleeve extruder whose screws have a diameter of 64 mm and a length equal to 29 times the diameter (extruder type

known as 29 D), the rotational speed of the screws may range from 80 to 100 rpm.

For a five-sleeve extruder in each case, the following table gives the temperatures for each of the sleeves.

Sleeve	Temperature
0	12°C
1'	60°C
2'	60°C
3'	80°C
4'	80°C
5'	80°C
Extrusion nozzle	70°C

After a residence time of 1 min 30 sec in the twin-screw extruder, the concentrate with high polymer content exits the extrusion nozzle at a temperature in the range of 100-120°C.

Figures 2a, 2b and 2c, which must be considered as arranged end-to-end, illustrate the treatments undergone in this case by the materials mixed in the five sleeves of the extruder. The abbreviations used in these diagrams have the following definitions:

TSF: transfer,

LM: low-shear mixing,

HM: high-shear mixing,

CM: counterthread mixing.

By applying the above conditions, the apparatus we have just described makes it possible to produce concentrates with high polymer contents having satisfactory elasticity for the preparation of chewing gums.

The concentrates thus prepared may thus have, depending on the intended use, a composition within the following composition range, in weight percent:

- high molecular weight elastomers: 30 to 85%
- low molecular weight elastomers: 0 to 5%
- rosin resin: 8 to 10%
- wax-type plasticizer: 2 to 10%
- mineral fillers: 5 to 10%

A gum base concentrate intended for the preparation of chewing gum tablets will preferably have the following composition in weight percent:

- elastomers: 85%
- mineral fillers: 18%
- plasticizers: 2%

The high molecular weight elastomers used in this case may have molecular weights in number Mn and in weight Mw which vary depending on the rotary speed of the five-sleeve twin-screw extruder's screw.

These weights will be, for example, as follows:

	Mn	Mw
— Continuous production at 80 rpm	135 900	208 800
— Continuous production at 100 rpm	131 800	200 000
— Continuous production at 120 rpm	128 800	196 400

For a concentrate intended for the preparation of bubble gum, a preferred composition will be as follows in weight percent:

- elastomers: 32%
- mineral fillers: 52%
- plasticizers: 16%

The high molecular weight elastomers used in this case may have the following characteristics as a function of the speed of the five-sleeve twin-screw extruder screw:

	Mn	Mw
— Continuous production at 80 rpm	86 500	459 700
— Continuous production at 100 rpm	79 700	609 000

In the concentrates thus prepared, the initial elastic properties of the elastomers are not altered by the preparation process and are practically unchanged in the finished product.

## Claims

1. Method of preparing a gum base with a high polymer content, of the type containing high molecular weight elastomers, plasticizers and mineral fillers, characterized by the fact that high molecular weight elastomers are ground to particles; these particles and the plasticizers are mixed in the desired proportions; the resulting mixture and the mineral fillers are introduced separately into a double-screw extruder (6) with jackets (0, 1', 2', 3', 4', 5'), in which the temperature does not exceed 120°C, where the elastomer mixture does not remain for more than about one minute and 30 seconds and which has a screw speed of less than about 120 rpm; and the concentrate exiting the extruder is collected.

2. Method according to Claim 1, characterized by the fact that the high molecular weight elastomers are ground to particles having a maximum size on the order of 5 mm.

3. Method according to one of Claims 1 and 2, characterized by the fact that the mixture of high molecular weight elastomers and plasticizers is introduced into the jacket (0) arranged the furthest upstream from the double-screw extruder (6), while the mineral fillers are introduced downstream from this mixture.

4. Method according to Claim 3, characterized by the fact that the mineral fillers are introduced into two different jackets (0, 2') of the double-screw extruder (6).

5. Method according to one of Claims 1-4, characterized by the fact that low molecular weight polymers are introduced in the molten state into the double-screw extruder (6) downstream from the mixture of high molecular weight.

6. Method according to one of Claims 1-5, characterized by the fact that the double-screw extruder (6) is a an extruder with five jackets (0, 1', 2', 3', 4', 5').<sup>1</sup>

7. Gum base concentrate prepared by a method according to Claims 1-6.

8. Gum base concentrate according to Claim 7, characterized by the fact that it contains at least 30% and preferably 85% by weight of polymer.

9. Use of a gum base concentrate according to one of Claims 1-8 for the manufacture of chewing gum.

---

<sup>1</sup> Text says "five jackets" but lists six numbers.—Tr.

1/2

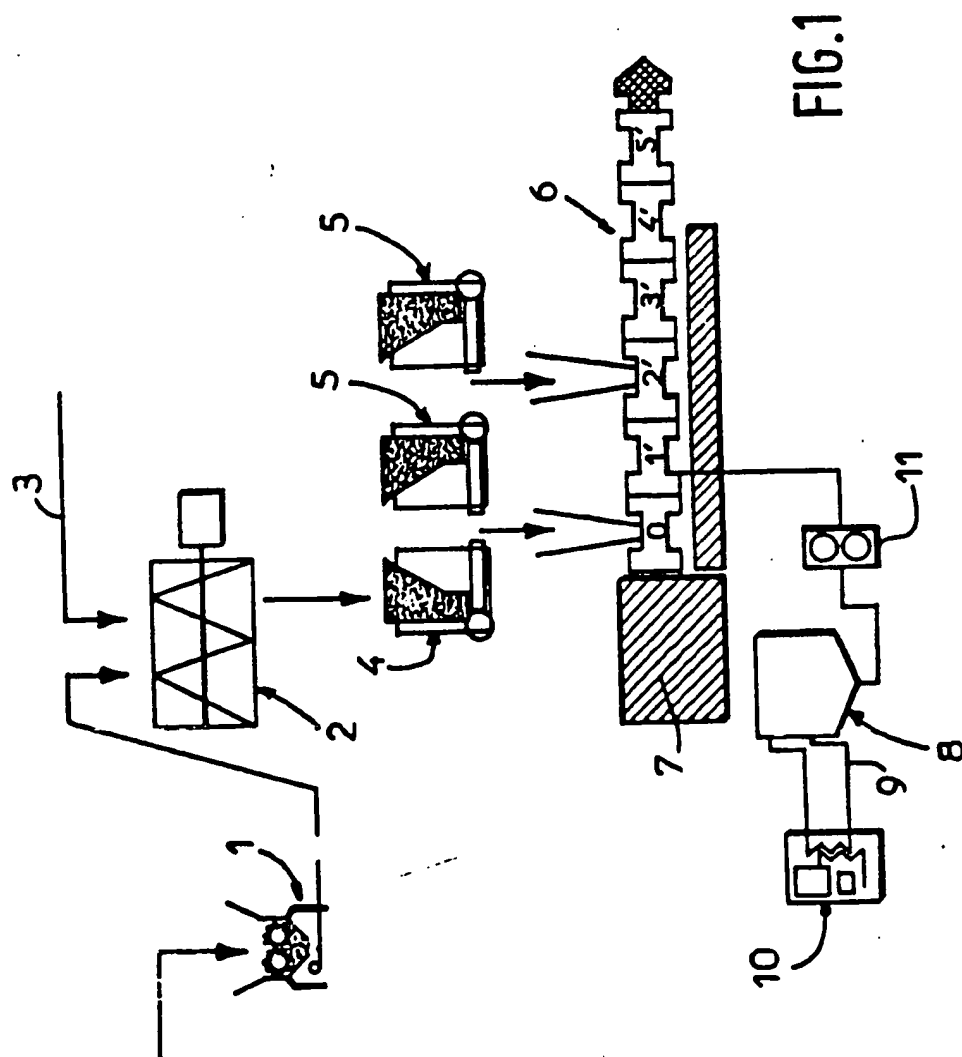


FIG. 1



